We claim:

1. A process for preparing 2-keto-L-gulonic C_4-C_{10}-alkyl esters by esterifying 2-keto-L-gulonic acid (KGA) with a saturated, branched or unbranched C_4-C_{10}-alcohol, which comprises, in a preliminary esterification, reacting an aqueous KGA solution with a C_4-C_{10}-alcohol under acid catalysis up to a degree of esterification of from 20% to 70% and dehydrating the product in a continuous rectification apparatus using a C_4-C_{10}-alcohol, as a result of which the esterification reaction advances.

2. A process as claimed in claim 1, wherein the alcohol is a saturated, branched or unbranched alkyl alcohol having from 4 to 10 carbons, preferably n-butanol.

3. A process as claimed in claim 1 or 2, wherein, in the preliminary esterification, the alcohol is used in a mass ratio to the KGA content in the aqueous solution of from 1:1 to 5:1.

4. A process as claimed in any of claims 1 to 3, wherein the catalyst is an acid heterogeneous or homogeneous catalyst.

5. A process as claimed in any of claims 1 to 4, wherein the catalyst is a mineral acid.

6. A process as claimed in any of claims 1 to 5, wherein the preliminary esterification is carried out in a continuous-flow stirred tank.

7. A process as claimed in any of claims 1 to 5, which is carried out under the following conditions:

   a) mean residence time of the aqueous KGA in the preliminary esterification from 1 to 3 h,

   b) reaction temperature in the preliminary esterification from 65°C to 120°C; and/or

   c) mass ratio of KGA content to C_4-C_{10}-alcohol from 1:1 to 5:1; and/or

d) reaction temperatures during the entire process from 50°C to 120°C and/or

e) use of from 0.02 to 0.03 mol of sulfuric acid per mole of KGA as catalyst.

8. A process as claimed in any of claims 1 to 7, wherein the aqueous KGA solution, before entry into the preliminary esterification reactor, is concentrated up to the solubility limit of KGA.

9. A process as claimed in any of claims 1 to 7, wherein the aqueous KGA solution, before entry into the preliminary esterification reactor, is concentrated to above the solubility limit of KGA.

10. A process as claimed in any of claims 1 to 9, wherein the continuous rectification apparatus (2) is equipped with an evaporator (3) and a condenser (4), and also preferably with a phase-separation apparatus (5) and/or a vacuum system (6).

11. A process as claimed in any of claims 1 to 9, wherein the preliminary esterification reactor (1) is equipped with an additional column (7), an additional evaporator (8) and an additional condenser (9) and also preferably with an additional phase-separation apparatus (10).

12. A process for preparing ascorbic acid, which comprises the process as claimed in any of claims 1 to 11 and the 2-keto-L-gulonic C_4-C_{10}-alkyl ester prepared being converted to L-ascorbic acid in one or more steps.

Process for preparing 2-keto-L-gulonic C_4-C_{10}-alkyl esters

Abstract

The invention relates to a process for preparing 2-keto-L-gulonic acid C_4-C_{10}-alkyl ester by esterifying 2-keto-L-gulonic acid (KGA) with a saturated, branched or unbranched C_4-C_{10}-alcohol, which comprises, in a preliminary esterification, reacting an aqueous KGA solution with a C_4-C_{10}-alcohol under acid catalysis to a degree of esterification of from 20% to 70%; and dehydrating the product in a continuous rectification apparatus using a saturated, branched or unbranched C_4-C_{10}-alcohol, as a result of which the esterification reaction advances. Preferably, the alkyl alcohol is n-butanol. In a preferred embodiment, the aqueous KGA solution is concentrated before the esterification up to the solubility limit or above, preferably under catalysis by a homogeneous or heterogeneous catalyst, in particular sulfuric acid, and at temperatures of from 50°C to 120°C. In a further embodiment, in one or more further steps, the KGA ester prepared is converted to L-ascorbic acid.